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TECHNICAL REPORT NO. 14

SOLID STATE EXTRUSION OF MELT-CRYSTALLIZED AND
SOLUTION-GROWN CRYSTALS OF NYLONS 6 AND 66

by

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SOLID STATE EXTRUSION OF MELT-CRYSTALLIZED AND
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ABSTRACT

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Melt and solution grown crystals of nylons 6 and 66 have been solid state extruded to examine the relationships among thermal behavior, crystallinity, orientation and modulus. Unextruded and extruded samples were examined by thermal analysis, wide angle x-ray and by tensile testing. An extrusion draw ratio (EDR) of 4.0 was attained for all samples. At this EDR, modulus and crystallinity are linearly related over a wide range up to 60% crystallinity. The highest tensile modulus of 6.7 GPa was attained for both polymers in extrudates of solution-grown crystals, although these extrudates of compacted precipitates were less oriented. This suggests that the extent of both crystallinity and draw play an important role in the enhancement of modulus. ↗

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INTRODUCTION

Solid state extrusion under selective conditions of high density polyethylenes crystallized from melt⁽¹⁾ and from solution⁽²⁾ has provided ultra-oriented morphologies of high tensile moduli. The semi-crystalline state of nylons 11 and 12 can also be solid-state extruded⁽³⁾. Nylons are thermally more stable than polyethylene due to inter- and intramolecular hydrogen bonding. The crystals and mechanics of nylons 6 and 66 are thermally yet more stable than in nylons 11 and 12⁽⁴⁾ due to a higher concentration of amide groups. They also have potentially high tensile moduli, comparable to polyethylene⁽⁵⁾. Higher crystallinity leads to higher moduli for melt-crystallized nylons⁽⁶⁾. Solution-grown nylon crystals⁽⁷⁻¹⁰⁾ have also been shown to exhibit a higher percent crystallinity⁽⁸⁾ than the corresponding melt-crystallized polymers.

In this study, both melt-crystallized and solution grown crystals of nylons 6 and 66 have been solid-state extruded through conical dies to examine the dependence of thermal behavior on extrusion draw ratio (EDR) and the relationships among modulus, crystallinity and orientation. The extrudates have been examined by thermal analysis, wide angle x-ray and by tensile tests.

MATERIALS AND EXPERIMENTS

Nylon 6 powder and cylindrical plugs of nylon 66 were obtained commercially. For determination of molecular weights, small amounts were solution purified by dissolving in formic acid followed by precipitation on pouring into water as by the method of Pennings⁽¹⁰⁾. The molecular weights were calculated to be 3.0×10^4 and 3.8×10^4 for nylons 6 and 66, respectively, from intrinsic viscosities of 1.07 and 1.41 in 85% and 90% formic acid.

Nylon 66 rods were melt-crystallized, as received. They were shaved to a diameter (ϕ) of 0.95 cm. Nylon 6 powder was melt-crystallized by slow cooling at a rate of $-1^\circ\text{C}/\text{min}$ under an applied pressure of -200 atm in the reservoir ($\phi=0.95$ cm) of an Instron Capillary Rheometer.

Both nylons were dissolved at several concentrations (0.1 - 25 w/w %) in 1,4-butanediol⁽¹⁰⁾ and then crystallized isothermally or by fast cooling. The suspensions obtained were poured into a 1:1 w/w mixture of methanol and acetone. The precipitates were dried in vacuo at 100°C over 48 hrs. and compressed at 120°C and 2400 atm in the Rheometer reservoir.

The prepared plugs were solid state extruded at several fixed temperatures and at constant extrusion rate; i.e., a fixed cross head speed of 0.2 cm/min through the stainless steel conical dies with an entrance angle of 20° and at several ratios of cross sectional areas for die entrance to exit, i.e., at several draw ratios, viz. 2, 3, 4 and 5. No lubricant, hydrostatic liquid nor plasticizer was used.

Thermal analysis of all samples was performed at a heating rate of 10°C/min in a Perkin-Elmer DSC model IB to estimate melting peak temperature (T_m) and percent crystallinity based on a reported⁽¹¹⁾ heat of fusion (ΔH_f) of perfect crystals (45 cal/gr) for both nylons.

Wide angle x-ray diffraction patterns were obtained with the use of nickel-filtered, copper K_α radiation.

Tensile moduli were calculated at a strain of 0.2% and at a strain rate of $3.33 \times 10^{-4} \text{ sec}^{-1}$ by using an Instron tensile testing machine, model TTCM, equipped with a 10 mm strain extensometer.

RESULTS AND DISCUSSION

Melt-Crystallized Nylons

Multi-melting temperatures for both nylons 6 and 66 have been reported and discussed by prior investigators⁽¹²⁻¹⁵⁾. Dumbleton et al.⁽¹³⁾ reported in nylon 66 that at increased annealing temperature and/or time, the temperature and area of the lower melting peak increased, while retaining the higher melting peak; but subsequently all could be transformed to the higher melting peak as the total crystallinity increased. Etching studies suggest an alternate explanation^(13a). The long period of crystallites also increased during this annealing process^(13,15).

Both nylons are known to exhibit polymorphism. Nylon 6 has α , β , and γ crystal forms, depending on preparation^(16,17); and nylon 66 exhibits α and β crystal forms⁽¹⁸⁾. It is well known^(4,16,18) that slow crystallization for both polymers produces the α form which is the most stable and does not convert to other forms on annealing or drawing. Slow crystallization was performed in this study on both nylons. Annealing effects at 180 and 200°C for 1 hour on the melting for unoriented nylons 6 and 66 are shown in Figures 1 and 2. Annealing increased the crystallinity and lower melting peaks but did not produce complete conversion of the lower melting peaks to the higher peaks, as reported at lower temperature and shorter annealing time. Results in Figures 1 and 2 are also representative of unoriented (unextruded) residual samples left in the capillary reservoir of the Instron Rheometer after a portion had been solid state extruded.

The extrudates of different EDR's were obtained in steady state after one hour by use of dies of different exit diameter. Melting behavior as seen in Figures 1 and 2 shows the effect of EDR at fixed extrusion temperatures (T_E). The higher melting peak for both nylons remained constant. On the other hand, the lower melting peaks increased with EDR and subsequently all was converted to the higher melting form. Crystallinity concurrently increased. These results suggest that the drawing effect on melting may be similar to that of annealing.

Solution-Grown Crystals

The solution-crystals of nylon 6 precipitated from 1,4-butanediol produced the α -form⁽¹⁰⁾, as confirmed in a subsequent figure, 8b. The solution-grown samples of nylon 6 were prepared by isothermal crystallization at 128°C from 0.1 and 1% solutions and by fast cooling to room temperature from 8 and 25 wt % solutions. Fast cooling crystallization was also performed on nylon 66 to obtain precipitates from 8 and 25 wt % solutions. This produced melting behavior different from melt-crystallized and from samples precipitated at lower concentrations, as seen in Figure 3. The lower T_m corresponds to 70% of the total area for the 8% solution-precipitate. This differs markedly from the shoulder or tail on the melting curves of the samples melt-crystallized and precipitated at lower concentrations. Nylon 6 precipitated from 0.1 and 1% solutions exhibited single melting peaks with a T_m of 222°C, the same as the higher T_m of the melt-crystallized sample.

Similar and more clear behavior was observed for nylon 66, as seen in Figure 4. The higher melting peak of solution-grown crystals, as shown in Figure 4, may be due to the melting of reorganized crystals formed during the DSC scan. The higher melting peak of the SGC may thus not correspond to the higher melting peak of the extruded samples, (b) in Figure 4^(13a). The larger area of the lower melting peak is associated with fast crystallization which produces smaller crystallites.

Precipitated and compacted nylon 6 and 66 were solid-state extrudable at 200 and 240°C to an EDR of 4.0. The limits of temperature and draw are likely due to crystallinity level, as shown in Table II. Extrudates of nylon 6 precipitate from 0.1 - 25% solutions exhibited single melting peaks. On the other hand, extrudates of nylon 66 showed double peaks with the lower peak only converted partially to the higher. Complete transformation of crystallites of nylon 66 likely require more extensive drawing since complete transformation was attained for nylon 6. The % crystallinity for both nylons is high after extrusion as seen in Table V.

Tensile Behavior of Extrudates

Conventional drawing effects on deformation^(19,20) and mechanical properties^(21,22) have been reported for these nylons. Higher draw provides higher orientation of both crystalline and amorphous regions. Nylons can absorb water in their amorphous component. However, the

tensile modulus of oriented nylon 66 conditioned at 50% RH is similar to that of dried samples, although the change from dryness to water saturation causes a four fold decrease in modulus because of the plasticizing action⁽²³⁾. Our experiments here were performed below 50% humidity. Thus, after preparation of solid plugs, sample drying was not performed in this study. Both crystallinity and tensile modulus increase with EDR, for both melt-crystallized nylon 6 and 66, as seen in Figures 5 and 6. The highest readily obtainable EDR was 4 for all samples, either melt-crystallized or precipitated. EDRs over 4 produced spiral fractures in extrudates. In comparable tests, nylon 12⁽³⁾ has been solid state extruded at EDR up to 12 without fractures and high density polyethylene⁽¹⁾ to an EDR of 36. The lower draw ratio obtained here for nylons 6 and 66 is likely due to high concentrations of amide groups leading to extensive interchain hydrogen bonding. Thus, the studies on deformation of nylons containing plasticizers with polar chemical groups have been reported to reduce yield stress and the work of deformation⁽²⁴⁾. Importantly, however, no high draw ratios for nylons 6 and 66 appear to have been reported. The highest moduli obtained for extrudates of melt-crystallized nylons 6 and 66 were 3.3 and 4.3 GPa, respectively. This nylon 66 extrudate is well oriented according to wide angle x-ray diffraction patterns, as seen in Figure 8a. A similar x-ray pattern was observed in nylon 6 extrudate.

Increase of crystallinity was observed with EDR for both nylons. The starting crystallinity for melt-crystallized nylons 6 and 66 is ~30%, as shown in Table I. Samples of both polymers annealed at 180 and 200°C

increased slightly in crystallinity. Crystallinity increased by ~20% on solid state extrusion to an EDR of 4 for both melt-crystallized polymers. On the other hand, the initial crystallinity for solution-grown nylons 6 and 66 is remarkably high, 50 - 60%, as shown in Table II. On solid-state extrusion, crystallinity stays high, increasing slightly. This suggests that ~60% may be near saturated for crystallinity of nylons 6 and 66. Extrusion of melt-crystallized nylon 66 at 220°C produced a higher modulus and crystallinity, due to annealing, than that at 200°C. This suggests that the higher crystallinity is responsible for a higher modulus. Figure 7 shows that relationship of tensile modulus to crystallinity at a fixed EDR of 4 for melt-crystallized and precipitated samples. The highest modulus, as obtained for both nylons, is 6.7 GPa which was for solution-grown samples. The linearity in Figure 7 is likely fortuitous, with extrapolation being unwarranted.

Extrudates of nylon 66 precipitate is less oriented than that of melt-crystallized samples, as seen in Figures 8-a and c; yet the modulus of the former is higher. Similar results were obtained for nylon 6. This indicates a dominant role of crystallinity in determining the tensile modulus of these solid-state extruded aliphatic nylons.

CONCLUSIONS

Solution-grown crystals of nylons 6 and 66 have been solid-state extruded. The thermal properties, crystalline orientation and tensile moduli of these extrudates are compared with solid-state extrudates of melt-crystallized nylons.

1. Of the dual melting peaks for each nylon, the concentration of crystallites having the highest melting isotherm was increased by solid-state extrusion.
2. The highest initial crystallinities for precipitates of 50 - 60% increased only slightly on solid-state extrusion to a draw ratio (EDR) of 4.
3. At an EDR of 4, the measured tensile moduli for both polymers increased linearly with crystallinity up to 60%.
4. Extrudates of precipitates for both polymers were less oriented at EDR of 4 than the corresponding melt-crystallized samples. The former nonetheless exhibited higher moduli. This indicates that crystallinity as well as drawing plays an important role in tensile modulus development.
5. The highest moduli obtained for extrudates of precipitates for each nylon 6 and 66 is 6.7 GPa. Extrudates of the same EDR of 4 for melt-crystallized samples of the same nylons 6 and 66 were 3.3 and 4.3 GPa.

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TABLE I: Crystallinity of Melt-Crystallized Nylons

Sample History	% Crystallinity		
	Nylon 6	Nylon 66	
Unextruded	28	29	
Extruded	at 180°C	at 200°C	at 220°C
EDR = 2	35	37	44
3	40	41	52
4	47	47	50
Unextruded residue in Rheometer	32	32	36

TABLE II: Crystallinity of Solution-Grown Nylons
and Their Solid-State Extrudates

wt % concentration	% Crystallinity			
	Nylon 6		Nylon 66	
	unextruded	extruded*	unextruded	extruded**
0.1	61	63	—	—
1.0	58	61	—	—
8.0	54	57	54	56
25	53	55	53	53

* at 200°C to EDR of 4

** at 240°C to EDR of 4

Uncertainty $\pm 1\%$ in crystallinity.

FIGURE CAPTIONS

- Figure 1: DSC melting behavior of melt-crystallized nylon 6: a, unextruded; b, unextruded and annealed at 180°C for 1 hr; c, d, and e, extruded at 180°C and at EDR of 2, 3 and 4, respectively.
- Figure 2: DSC melting behavior of melt-crystallized nylon 66: a, unextruded, b, unextruded and annealed at 220°C for 1 hr; c, d and e, extruded at 220°C and at EDR of 2, 3 and 4, respectively.
- Figure 3: Melting behavior of nylon 6 precipitates from 8% solution: a, unextruded; b, extruded at 200°C at EDR of 4.
- Figure 4: Melting behavior of nylon 66 precipitates from 8% solution: a, unextruded; b, extruded at 240°C and at EDR of 4.
- Figure 5: Increase in tensile modulus and crystallinity with extrusion draw ratio for melt-crystallized nylon 6 extruded at 180°C: ●, tensile modulus, ★, crystallinity.
- Figure 6: Increase in tensile modulus and crystallinity with extrusion draw ratio for melt-crystallized nylon 66: ● and ★, tensile modulus and crystallinity for extrudates at 200°C (filled symbols), for extrudates at 220°C (open symbols).
- Figure 7: Relationship of tensile modulus to crystallinity for nylons 6 and 66 at a fixed draw ratio of 4: ●, nylon 6; ★, nylon 66.

Figure 8: Wide angle x-ray diffraction patterns for nylon 66: a, melt-crystallized sample extruded at 220°C and EDR of 4; b, 8% solution grown sample unextruded; c, 8% solution-grown sample extruded at 240°C and EDR of 4.

Figure 1

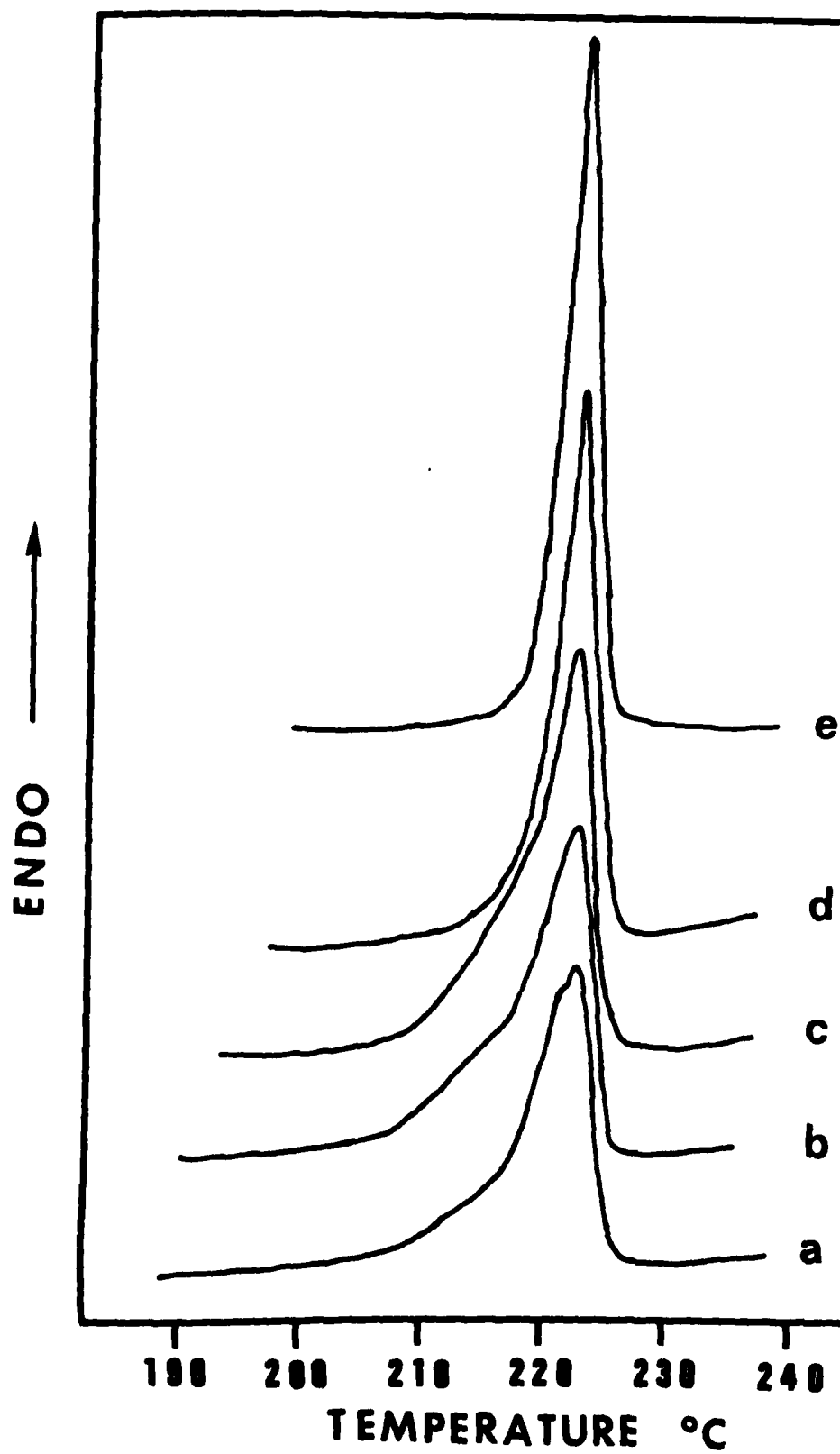


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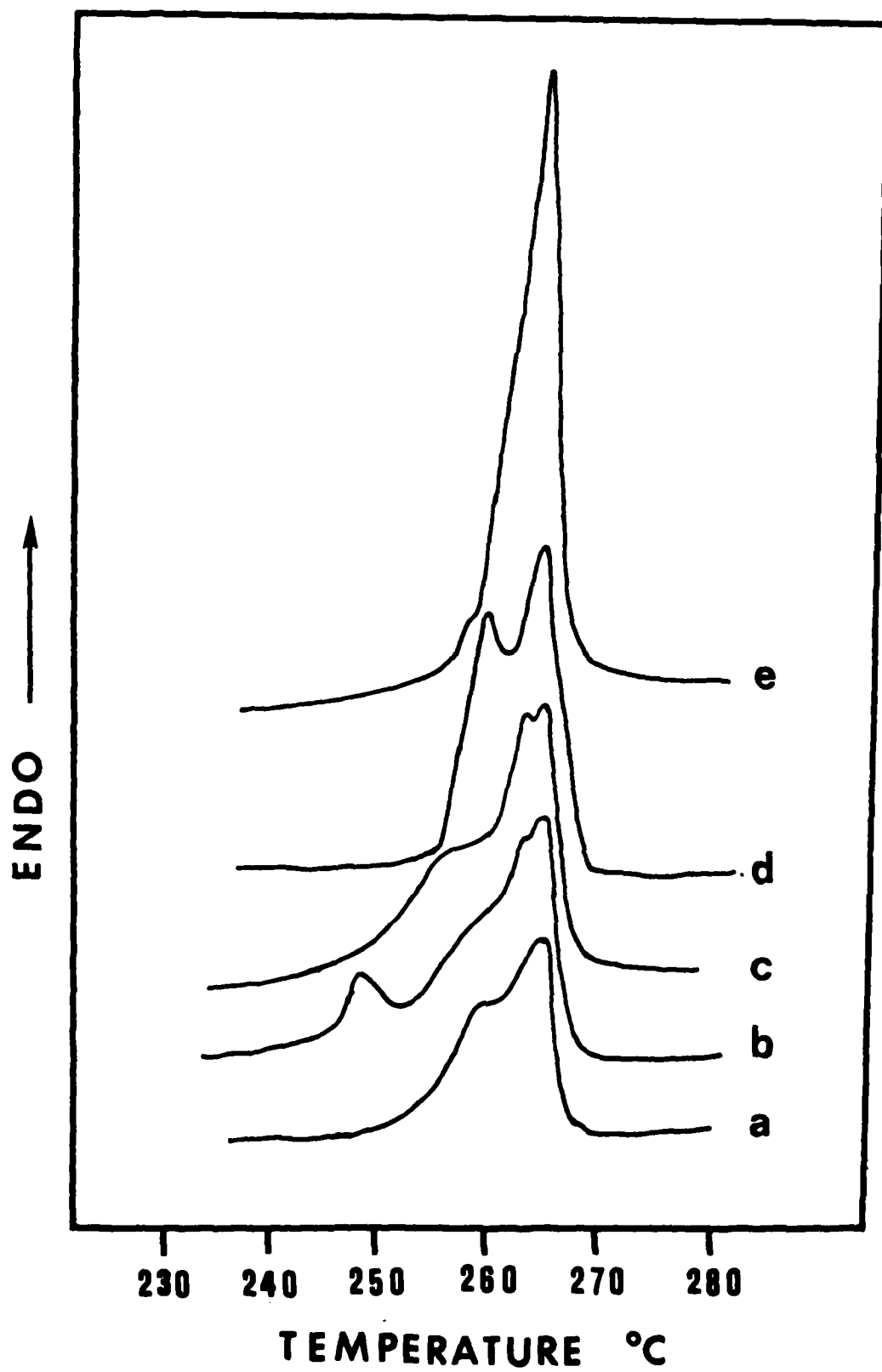


Figure 3

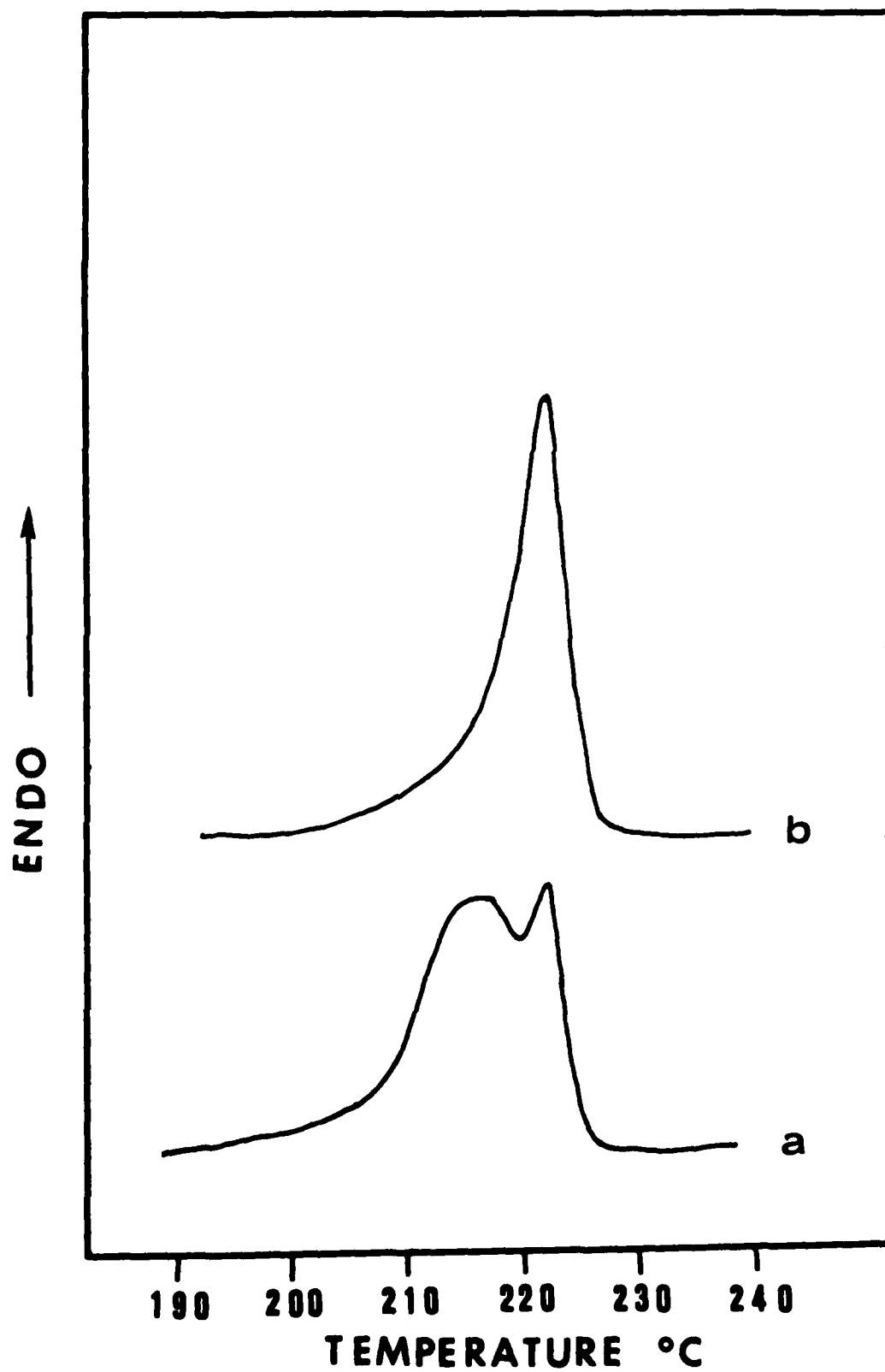


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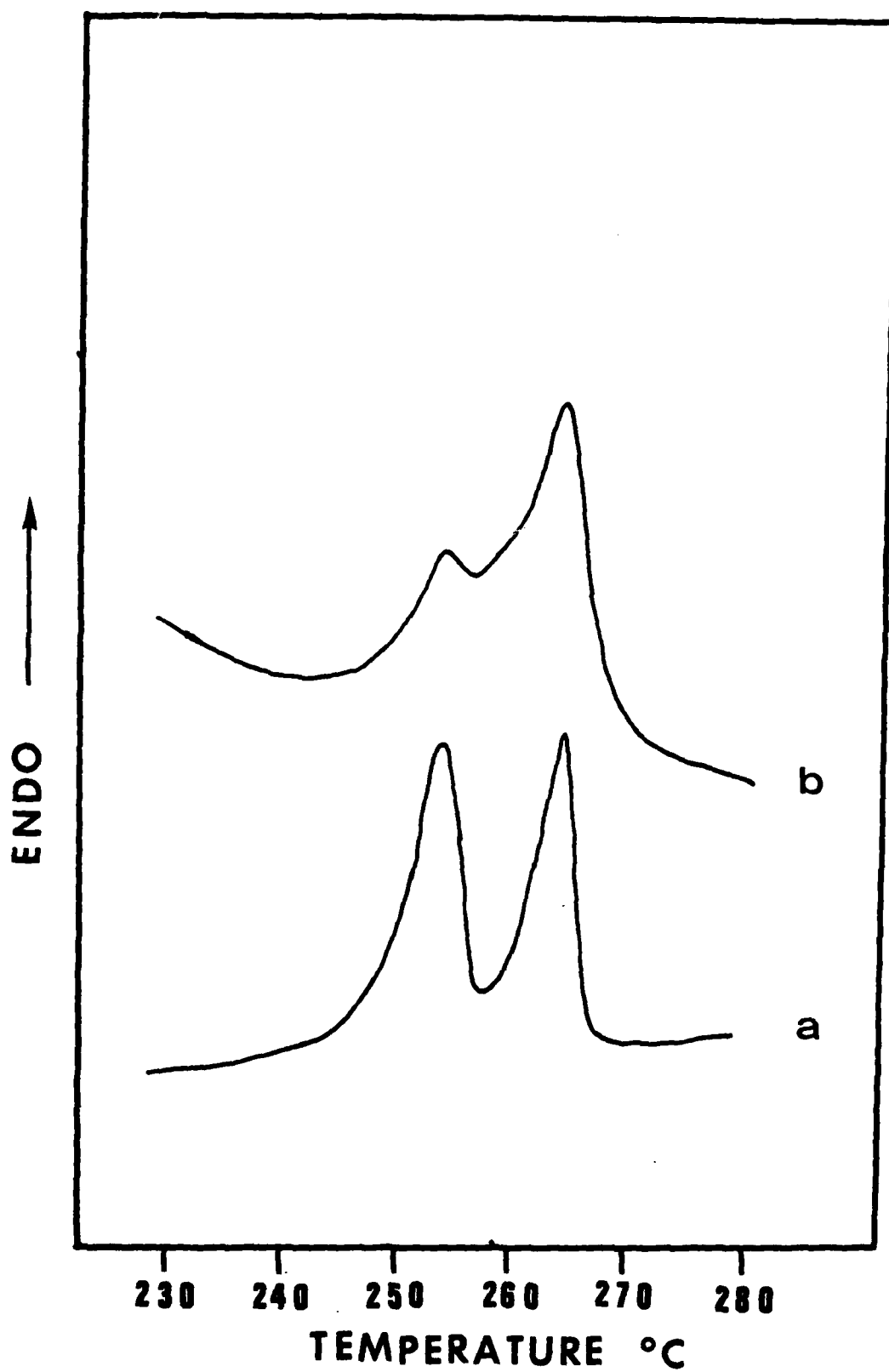


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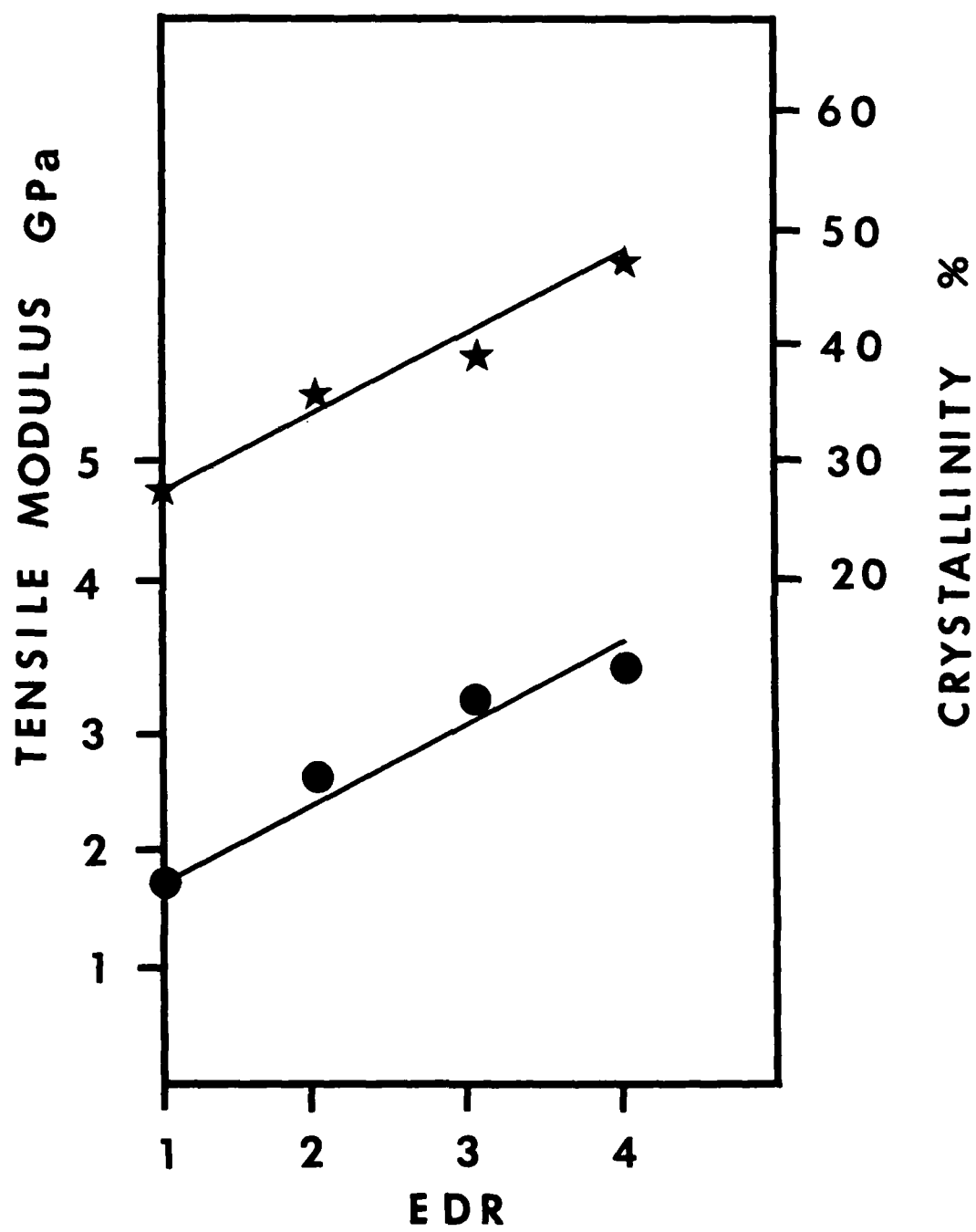


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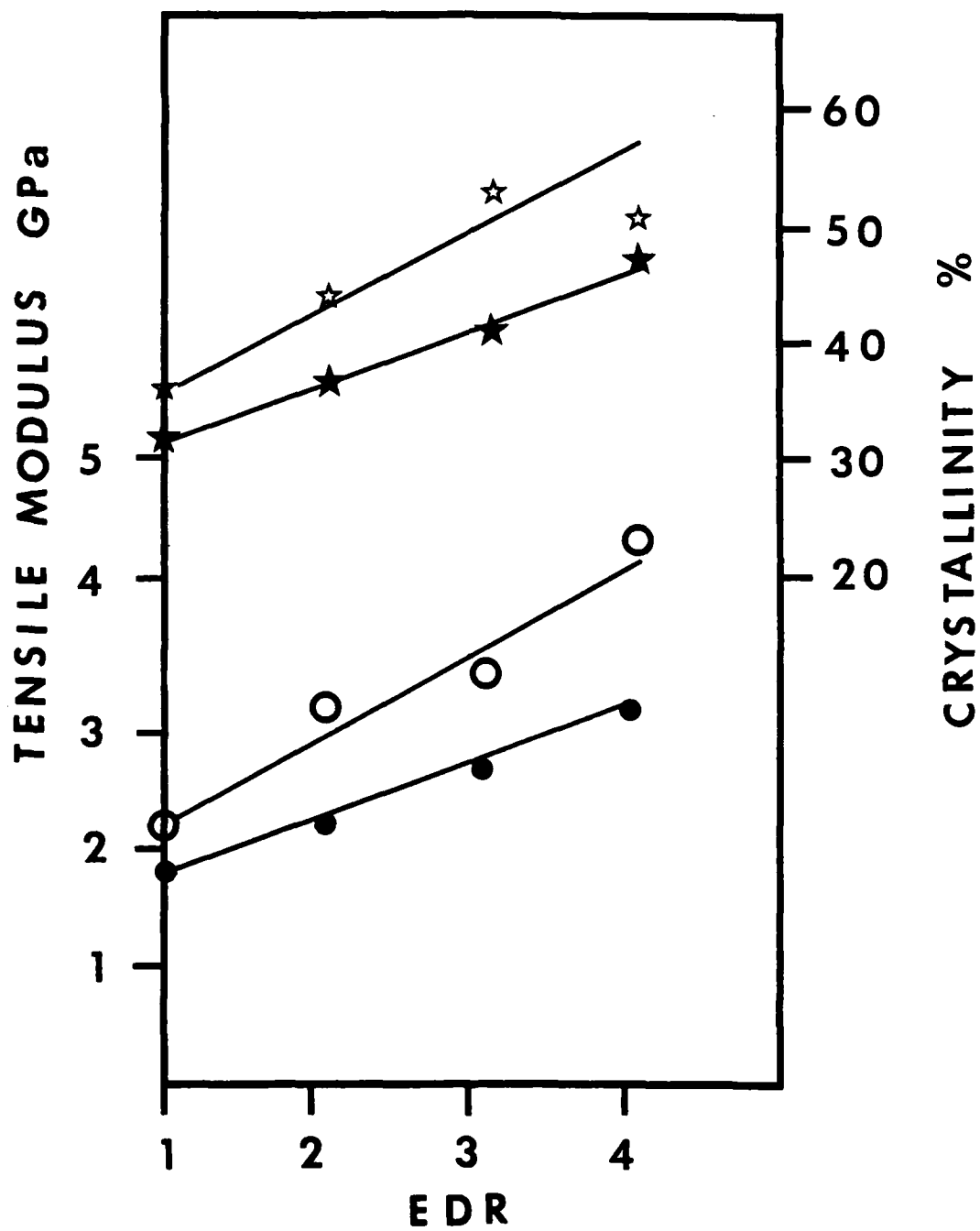


Figure 7

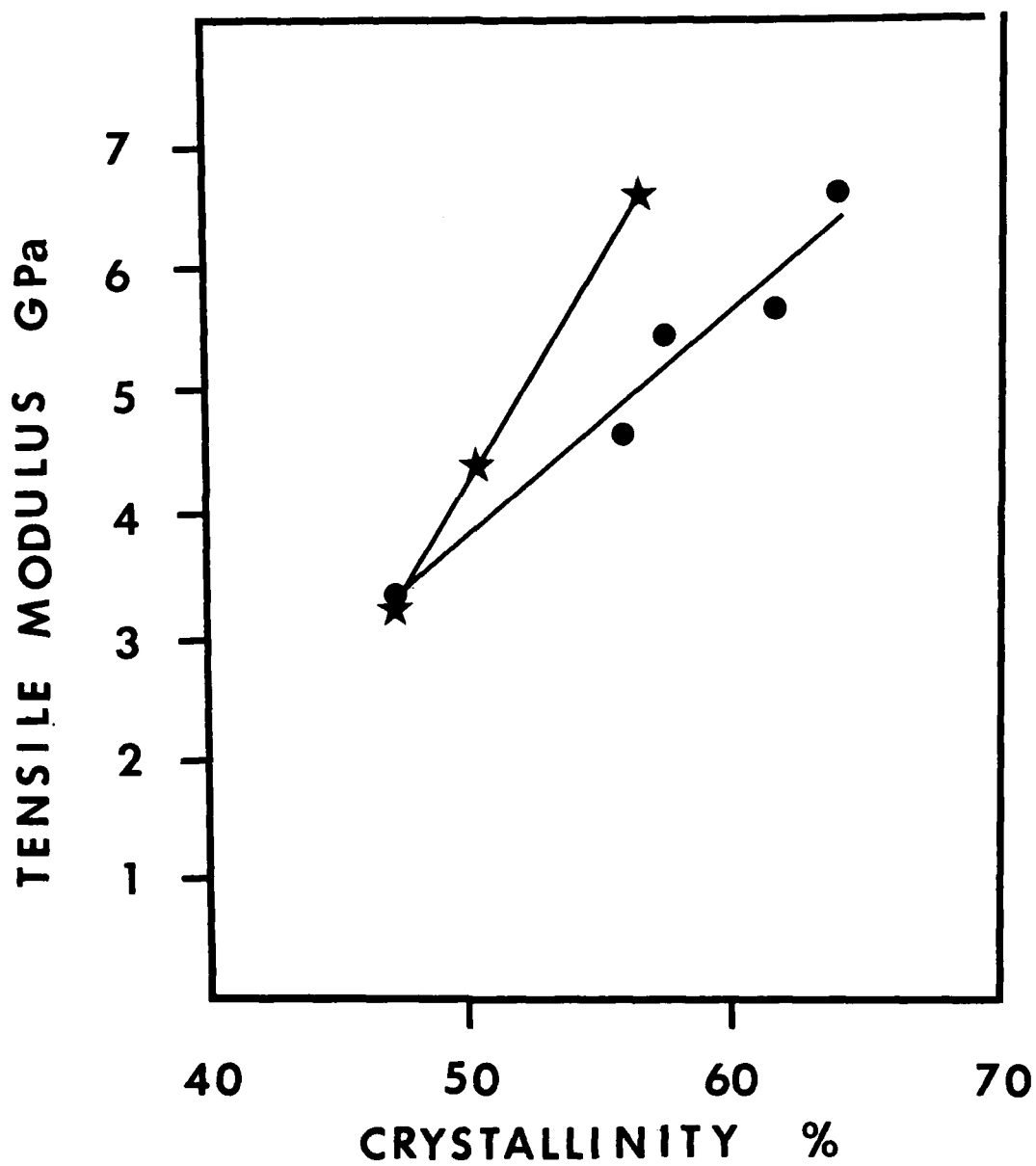
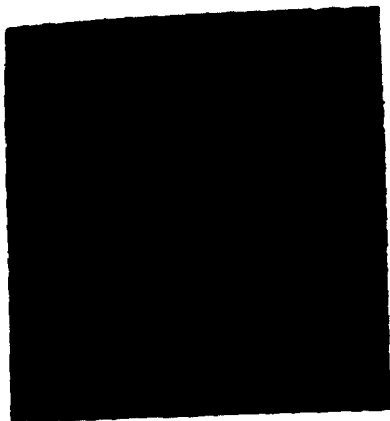
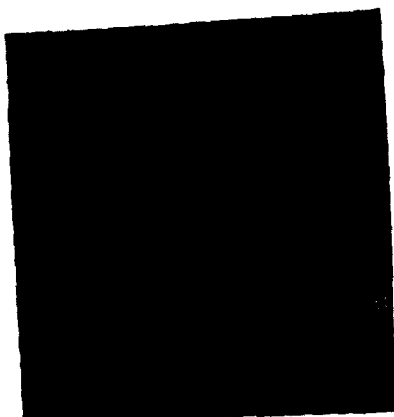


Figure 8

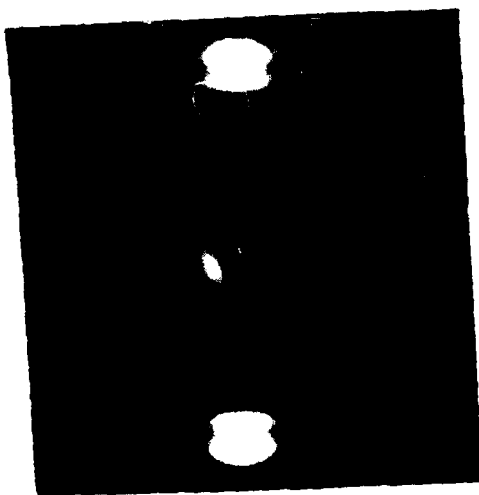
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